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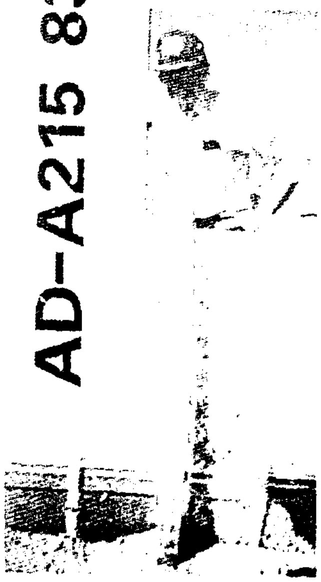

REFERENCE PROPERTIES OF CEMENT-BASED PLUGGING AND SEALING MATERIALS FOR THE WASTE ISOLATION PILOT PLANT (WIPP)

by

Charles W. Gulick

Sandia National Laboratories
Albuquerque, New Mexico 87185

and

Lillian D. Wakeley

Structures Laboratory

DEPARTMENT OF THE ARMY
Waterways Experiment Station, Corps of Engineers
3909 Halls Ferry Road, Vicksburg, Mississippi 39180-6199

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The Structures Laboratory of the US Army Engineer Waterways Experiment Station has conducted research on cement-based composites for the Waste Isolation Pilot Project (WIPP) since 1977 in support of Sandia National Laboratories. Sealing shafts and plugging boreholes for closure of this geologic repository for radioactive wastes will require grouts and concretes. Salt-saturated grouts and concretes bond adequately to the bedded halite host rock. Fresh-water mixtures will plug and seal penetrations through nonhalite strata.

This report gives reference values for such properties as unconfined compressive strength, static modulus, restrained expansion, and permeability of candiate grouts and concretes. Values tabulated here are not averages but are values that one could reasonably expect to attain with other candiate mixtures of similar composition. They are values that the authors consider reasonable targets for cement-based composites likely to be used in the closure phase of operation of the WIPP or other underground repository.

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Preface

The work described in this report is part of an ongoing research effort accomplished in the Concrete Technology Division (CTD), Structures Laboratory (SL), US Army Engineer Waterways Experiment Station (WES), under contract to Sandia National Laboratories (SNL), Albuquerque, NM. Dr. E. J. Nowak was Technical Monitor for SNL during preparation of this report, completed in the CTD under SNL Document No. 63-5609. Mr. Charles W. Gulick was the SNL Technical Monitor during most of the field and laboratory work that contributed to this study.

This report draws on observations and data collected between 1975 and 1988 from both laboratory and field activites that involved many members of the CTD staff, especially those in the Grouting and Petrography Units. Most of that work was directed or accomplished by Mr. Donald M. Walley. Messrs. John A. Boa, Jr., and Alan D. Buck, and Dr. Lillian D. Wakeley also worked extensively on many aspects of the project.

Mr. Bryant Mather was Chief, SL, throughout the period of this work. Preparation of this report was under the general supervision of Mr. Kenneth L. Saucier, Chief, CTD; Mr. Richard L. Stowe, Chief, Materials and Concrete Analysis Group, CTD; and Mr. James T. Ballard, Assistant Chief, SL. Dr. Wakeley was Principal Investigator for WIPP-related work during preparation of this report that she accomplished with assistance from Messrs. Gulick and Walley. The report was prepared by Mr. Gulick and Dr. Wakeley.

COL Larry B. Fulton, EN, is Commander and Director of WES. Dr. Robert W. Whalin is Technical Director.

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Conversion Factors, Non-SI to SI (Metric) Units of Measurement

Non-SI units of measurements used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
cubic feet (ft^3)	0.028318685	cubic metres (m^3)
cubic yards (yd^3)	0.7645549	cubic metres (m^3)
microdarcy	9.869333E-13	square metres (m^2)
pounds (force) per square inch (psi)	6.894757	kilopascals (kPa)
pounds (force) per square inch (psi)	0.006894757	megapascals (MPa)
pounds (mass)	0.4535924	kilograms (kg)
thousands of pounds (force) per square inch (ksi)	6.894757	megapascals (MPa)

REFERENCE PROPERTIES OF CEMENT-BASED PLUGGING AND SEALING MATERIALS FOR THE WASTE ISOLATION PILOT PLANT (WIPP)

Purpose

1. This report summarizes the development of cementitious materials for use at the Waste Isolation Pilot Plant (WIPP) site. Development and reference properties of two grouts and one concrete which have been used at the WIPP site are reported for guidance in the design of additional experiments and facilities. The reference values reported herein are based on the extensive laboratory testing of grouts and concretes from both laboratory and field activities, and are representative rather than average values. Actual values from these tests have been presented in previous reports, which are cited in this document.

2. The two grouts for which reference properties are given are BCT-1FF, a freshwater grout, and BCT-1F, a saltwater grout. The concrete is ESC (Expansive Salt Concrete). These grouts and concretes represent what was learned from a decade of experiments and field experience, before and in support of the WIPP Plugging and Sealing Program (PSP). Thus, their properties are offered as reference values, for guidance in any future materials-development programs for salt repositories. This was the current state of development of candidate cement-based seal materials for the WIPP, when this report was prepared. Properties anticipated for freshwater concrete to be used in future testing at the WIPP also are included.

Background

3. The US Department of Energy is developing the Waste Isolation Pilot Project (WIPP) in southeast New Mexico to provide a research and development (R&D) facility to demonstrate the technology for safe disposal of radioactive wastes resulting from defense programs of the United States (Public Law 96-164).

4. A part of the WIPP R&D program conducted by Sandia National Laboratories (SNL) is the Plugging and Sealing Program (PSP). The PSP is an

integrated program of modeling, laboratory materials testing and in situ tests to develop acceptable sealing technology for the operation and eventual decommissioning of the WIPP facility. Included in the objectives of the PSP is the development and assessment of long-term geochemical and mechanical stability of candidate seal materials (Stormont 1984).

5. Sealing design concepts have been presented for WIPP penetrations (Christensen, Gulick, and Lambert 1983; Stormont 1984). These concepts include the use of cement-based materials (grouts and concretes), and "natural" materials (salt and bentonite). Bulkhead-type seals in the shafts and at the entries to waste disposal panels are primary features of the conceptual seal designs. The bulkhead-type seals may be comprised of concrete, or of block components using bentonite and mined-out salt. The designs also include sealing boreholes with cementitious grouts.

Development of Grouts and Concretes for the WIPP

6. Research on and development of cementitious materials for the WIPP has been underway at the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station (WES) since 1975. Early work involved extensive laboratory studies at WES and discussions with technical employees of several oil well cementing service companies. A durable grout with low permeability was needed for placing plugs in a drill hole at the WIPP site for the Bell Canyon Tests in 1979 and 1980.

Freshwater grout (BCT-1FF)

7. A freshwater grout (BCT-1FF) was developed for mixing and placement in 1977 by a service company under WES supervision (Table 1). Grout plugs were placed in boreholes through anhydrite underlying the salt beds. The coarse grained (Class H) cement with low C_3A content, available in the WIPP site region, was selected as the main cementitious component of this grout. Fly ash was included in the mixture to reduce heat evolution during early hydration, and for durability. A high-range water-reducing admixture was used to reduce the water-to-solids ratio, and thus increase density and decrease permeability. The hemihydrate (plaster) form of calcium sulfate was added to form expansive phases in the hardened plugs. Specimens cast in the field during these early grouting operations were stored and tested at the WES lab in

Vicksburg at increasing ages (Gulick, Walley, and Buck 1980; Gulick, Boa, and Buck 1980 and 1982; Wakeley, Walley, and Buck 1986).

8. In late November 1981, the steel liner for a shaft designated as the Site and Preliminary Design Validation (SPDV) shaft (and subsequently the personnel access and salt-handling shaft) was grouted into place. The lower portion of the annulus between the liner and the rock was filled with about 5,700 ft^{3}* (161 m^{3}) of freshwater grout, closely related to the BCT-1FF formulation, in less than six hours. This large volume operation demonstrated quality control procedures for successfully batching and mixing the freshwater grout. Specimens were cast and shipped to the WES Structures Laboratory (SL) for testing and evaluation. Physical properties, phase compositions, and microstructure were comparable to the earlier placements of the BCT-1FF grout (Wakeley, Walley, and Buck 1986).

Saltwater grout (BCT-1F)

9. In 1983, the B-25 exploratory drill hole at the WIPP was plugged. For the lower halite zone, a saltwater grout (BCT-1F) was developed by adding enough fine granulated salt to the freshwater mixture proportions to saturate the mixing water (Table 2). Saturation with salt was expected to improve bonding between grout and rock, by preventing the grout mixing water from dissolving the halite host rock. Laboratory tests proved that this modification was effective in improving the bond at the interface between grout and rock.

10. Freshwater (BCT-1FF) grout was used for the upper part of the hole through the nonhalite and water-bearing zones. Again, specimens of both grouts were cast and shipped to the WES SL for testing and evaluation (Wakeley, Walley, and Buck 1986).

11. During September 1984, the first emplacement of the Plug Test Matrix was completed. The saltwater grout (BCT-1F) was mixed underground at WIPP and pumped into vertical holes in the floor of rooms L1 and L2 (Figure 2 of Stormont 1986). The purpose of this and later emplacements was to cure various candidate seal materials *in situ* in the WIPP environment. Periodic recovery of samples of these plugs was planned, to evaluate the reactions and interactions which occurred between the plug material and the host rock.

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* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page 3.

These materials were still in place and available for testing, at the end of FY89.

Expansive salt-saturated concrete (ESC)

12. In 1985, planning for plugs to be emplaced for Series A of the Small Scale Seal Performance Tests (SSSPT) required the development of an expansive concrete at WES. These field experiments involved early-age testing of permeability along the interface between host rock and salt-saturated concrete, and of temperature, strain, stress, and displacement of seals relative to the adjacent rock (Stormont 1986, 1987). Previous formulations of salt concrete, with the same cementitious system as the BCT freshwater and saltwater grouts, had not been as expansive as was judged necessary for the scheduled instrumentation and testing. Increasing the expansivity of the concrete was considered to be the best assurance of a very tight seal from a very early age to enhance the planned testing.

13. Development of expansive salt-saturated concrete (ESC, Table 3) was accomplished by June 1985 (Wakeley and Walley 1986). Concretes developed in the laboratory at WES prior to ESC had exhibited far less expansion than grouts based on the same cementitious system (Buck 1985), due in part to the large volume of aggregate in the concrete. Because the aggregate was necessary to reduce early heat evolution, improve physical properties, and reduce cost, it was necessary to increase expansivity of the nonaggregate portion of the concrete. An expansive admixture, marketed at that time by Master Builders as ChemComp III (CCIII), was added to the cementitious system modified from the BCT grouts to achieve the desired expansivity of ESC. The resulting 10-component concrete includes large amounts of calcium sulfate and calcium aluminosulfate to promote chemical expansion at an early age.

14. Sodium chloride (NaCl) adequate to saturate the mixing water was dry-batched with the cementitious solids of ESC to prevent dissolution of the host rock during placement and hardening. Other components of ESC were chosen to enhance various aspects of its rheology and early-age behavior. Coarse-ground oil well cement hydrates slowly which increases working time, and helps to decrease early heat evolution. The Class C fly ash used not only contributes compounds essential to the formation of expansive phases, but also increases workability and cohesion in this low-water but pumpable and self-leveling mixture. Sodium chloride and sodium citrate act as retarders for the

portland cement and calcium sulfate components respectively, resulting in a working time (without significant slump loss) of over 3h, and initial time of setting of more than 9h. The fine and coarse aggregates in ESC are commercially available near the WIPP site to reduce cost.

Proposed freshwater concrete

15. A field event involving placement of an expansive concrete, without salt but chemically tailored to other demands of the WIPP, had been planned for 1988. The concrete to be used for this work has not been formulated, nor had any field tests of freshwater concrete been accomplished by the end of FY89. However, there has been extensive laboratory development and testing at WES of concrete mixtures for repository sealing, the data from which will be adapted to this need. The formulation is expected to be similar to that for the salt-saturated concrete (Table 4), with substitution of a high-range water-reducing admixture and set retarder for the sodium chloride and sodium citrate.

Reference Properties of WIPP Grouts and Concretes

16. Table 5 gives reference property values for freshwater grout based on BCT-1FF. Additional data and discussion of this grout were reported by Gulick et al. (1980, 1981), Roy et al. (1985), and Wakeley et al. (1986). Reference properties for the BCT-1F saltwater grout are in Table 6. Other background information about the Bell Canyon Tests appear in several reports listed in Appendix A.

17. Reference properties for the expansive salt-saturated concrete tested in the field in 1985 and 1986 are in Table 7. Wakeley and Walley (1986), and Wakeley (1987 a,b) described development and properties of this concrete. Details of the field tests for which it was used are given by Stormont (1985, 1987), and Stormont and Howard (1986).

18. Proposed reference properties for a freshwater concrete are in Table 8. Buck (1985) and Wakeley and Roy (1985) present data from laboratory-cast concretes to which the field concrete is expected to be similar.

19. Sample sizes and types and details of test methods used to determine the measured physical properties from which these composite values are derived appear in many previous reports (see references).

Discussion

20. Between 1979 and 1986, there were seven field events at the WIPP involving WES in placement of saltwater (three) or freshwater (two) grouts, or both (two events); and two field placements of salt-saturated concrete. During this time, the grout formulations underwent some modification and improvement with the result that somewhat different but closely related grout formulations were actually placed in the field for each grouting event.

21. Variability in properties among sets of field-cast specimens from separate grouting events commonly results from the usual field variables of batch size, temperature, supplier and operator differences in batching, mixing, and placing, and multiple batches or lots of each component. Additional variability was introduced into the measurable properties of freshwater and salt-saturated grout by the above-mentioned slight modifications in formulation. Further, types and sizes of specimens cast in the field and tested in the laboratory, and conditions of curing and storage changed over the years in response to perceived data needs, available test equipment, and personnel. This also contributed to the variability in measured properties for grout summarized here as simply BCT-1F or BCT-1FF grout.

22. The ESC placed in 1985 and 1986 was the same formulation both times and batching and mixing procedures were kept as constant as practicable for the two field events. However, in the first case (SSSPT Series A), vertical boreholes were filled by free fall or tremie placement of the concrete. In the second event (SSSPT Series B), a concrete pump was used to fill horizontal boreholes. Specimens for laboratory tests also were cast of concrete that had been through the pump. And again, the types and sizes of specimens cast in the field for laboratory tests changed in response to perceived needs, yielding data from specimens of different configuration and conditions of curing and storage.

23. Physical and mechanical properties have been determined in the laboratory on specimens both cast in the laboratory and cast in the field (Buck, 1985; Wakeley, Walley, and Buck 1986; Comes, Wakeley and O'Neil, 1987). Laboratory curing and storage conditions for specimens from most of the field placements were planned to simulate at least some aspects of field conditions. Because of the long time period covered by development and placement of these mixtures, and changes in many conditions during that time, no single condition

or specimen configuration is chosen as the reference condition for the grouts or concretes. Instead, the values given for properties in Tables 5 through 8 are composite values derived from trends observed in spite of the variability. This avoids the limitation of defining a standard reference specimen type or size for each property, which would limit the usefulness of these recommended values to future experimental and demonstration programs. They are intended to be target values for properties of cement-based materials used in these programs.

Conclusions

24. The properties required of grouts and concretes developed for and used at the WIPP have evolved over time as successive formulations have benefited from further research. Nonsalt mixtures were designed for minimum permeability and long-term durability. Salt-based mixtures first met the requirement of avoiding dissolution of water-soluble host rock. They were then refined and modified for minimum permeability, durability, increased expansion, and long working time for ease of placement. All of these requirements were met, culminating in the expansive salt-saturated concrete, which also achieved high strength and use of aggregate readily available near the WIPP Site.

25. Cumulative experiences of research and development programs on cement-based materials for the WIPP now permit definition of target values for physical and mechanical properties of these materials. The usefulness of cement-based grouts and concretes as components of a comprehensive, multiple-barrier seal system for the WIPP, or for any radioactive-waste repository in bedded halite rock has been demonstrated.

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Table 1

Components and Proportions of Freshwater Grout (BCT-1FF)

Component	% of Total by mass	% of Total Solids by mass	Batch Weight for 1 ft^3, lb
Class H Cement	53.1	67.8	69.8
Class C Fly Ash	18.1	23.1	23.8
Cal Seal (Plaster)	6.5	8.2	8.5
Dispersant	0.68	0.9	0.9
Defoamer	0.02	0.02	0.02
Water	21.6	-	28.3
	100.0	100.02	131.32

Table 2

Components and Proportions of Salt-Saturated Grout (BCT-1F)

Component	% of Total by mass	% of Total Solids by mass	Batch Weight for 1 ft^3, lb
Class H Cement	48.3	61.2	61.9
Class C Fly Ash	16.2	20.6	20.8
Cal Seal (Plaster)	5.7	7.2	7.3
Salt (NaCl)	7.9	10.0	10.1
Dispersant	0.78	1.0	1.0
Defoamer	.02	.02	.02
Water	21.1	-	27.1
	100.0	100.02	128.22
			(101.12)

Table 3

Components and Proportions of Expansive Salt-Saturated Concrete (ESC)

	% of Total by mass	% of Total Solids by mass	Batch Weight for 1 yd^3, lb
Class H Cement	9.03	9.66	365
Chem Comp III	6.02	6.45	243
Cal-Seal	1.80	1.94	73
Class C Fly Ash	5.10	5.44	205
Fine Aggregate	34.11	36.50	1,365
Coarse Aggregate	34.58	37.00	1,381
NaCl	2.50	2.65	100
Defoaming Agent	0.21	0.24	9
Na Citrate	0.11	0.12	4.4
Water (Iced)	6.60	-	292
	100.06	100.00	4,037.4

Table 4

Components and Proportions of Proposed Freshwater Concrete

Component	% of Total by mass	% of Total Solids by mass	Batch Weight for 1 yd^3, lb
Class H Cement	15.3	16.4	600
Cal-Seal (Plaster)	1.9	2.1	75
Class C Fly Ash	5.1	5.5	200
Fine Aggregate	35.7	38.2	1,400
Coarse Aggregate (3/4" MSA)	35.2	37.6	1,380
Dispersant	0.2	0.3	10
Water	6.6	-	260
	100.0	100.1	3,925

Table 5

Recommended Mechanical Properties of Freshwater Grouts

Based on BCT-1FF Grout

	28 days	90 days	1 year
Unconfined Compressive 4x8" Cylinders			
Ksi	11.5	13.0	20.0
MPa	79.2	89.6	137.8
Static Modulus of Elasticity			
10^6 psi	3.1	3.5	3.8
10^3 MPa	21.4	24.1	26.2
Restrained Expansion, %	0.10	0.12	0.15
Permeability to Water, Microdarcy	0.9	0.9	0.9

Table 6

Recommended Mechanical Properties of Saltwater Grouts

Based on BCT-1F

	28 days	90 days	1 year
Unconfined Compressive Strength			
Ksi	7.2	9.9	11.9
MPa	49.6	68.0	82.0
Static Modulus of Elasticity			
10^6 psi	1.9	2.9	3.0
10^3 MPa	13.1	20.0	20.7
Restrained Expansion, %	0.05	0.11	0.38
Permeability to Brine, Microdarcy	<1.0	1.0	1.0

Table 7

Recommended Mechanical Properties of Saltwater Concrete

Based on ESC

	28 days	90 days	1 year
Unconfined Compressive Strength, 4x8" cylinder f'_c			
Ksi	4.5	5.0	6.9
MPa	31.0	34.5	47.5
Static Modulus of Elasticity			
10^6 psi	3.0	3.5	4.7
10^3 MPa	20.7	24.1	32.4
Poissons Ratio	0.20	0.19	0.19
Restrained Expansion, %	0.09	0.13	0.21
Permeability to Brine, Microdarcy	<0.8	0.8	0.8

Table 8

Recommended Properties of Freshwater Concrete

	28 days	90 days	1 year
Unconfined Compressive Strength			
Ksi	10.0	11.7	12.5
MPa	69	80	86
Static Modulus of Elasticity			
10^6 psi	4.8	5.1	5.2
10^3 MPa	33	35	36
Poissons Ratio	0.17	0.16	0.15
Restrained Expansion, %	0.03	0.05	0.05
Permeability to Brine, Microdarcy	<0.5	0.5	0.5